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DESIGN DATA STUDY FOR COATED COLUMBIUM ALLOYS

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Materials Processing Department
THOMPSON RAMO WOOLDRIDGE INC.
23555 Euclid Avenue
Cleveland 17, Ohio

TM 3710-67

ABSTRACT

A brief resume' is presented of the design data study of Pfaudler and TRW coated 30 mil FS-85 alloy sheet recently completed under Bureau of Weapons Contract NOw 62-0098c. A high carbon level in this material (250 ppm) was considered to be a contributing factor to a pronounced strain aging behavior and notch sensitivity of this alloy evidenced by tensile tests in the 1200-1600°F temperature range. A second lot of material, processed to specification, was acquired for additional tensile tests at room temperature and 1200°F. Smooth and notched tensile properties are reported for uncoated and TRW coated FS-85 alloy sheet from this lower interstitial heat. The notch sensitivity and influence of the protective coatings on the fracture behavior of this alloy are discussed.

Data is also presented on the cyclic oxidation to failure of prestrained coated specimens used in the previous program to establish the prestrain tolerance of the Pfaudler and TRW coatings. Retention of 2 hour oxidation protection at 1600 and 2600°F, after elastic and plastic tensile deformation, had previously been utilized as the criteria for determining the coating prestrain tolerance.

The current Design Data Study for coated columbium alloys is discussed in terms of the mechanical properties which will be evaluated, the evaluation techniques and the proposed modifications of previously utilized test methods. Mechanical properties of four coating-base metal systems involving the Pfaudler and TRW coatings applied to X-110 (Cb-10W-1Zr) and B-66 (Cb-5Mo-5V-1Zr) alloys will be investigated in this program.

DESIGN DATA STUDY FOR COATED COLUMBIUM ALLOYS

1. INTRODUCTION

Advancements in the development of reliable coating systems for the protection of columbium base materials continue to stimulate confidence in the utilization of coating-columbium base metal systems in aerospace structural and heat shield applications. However, there are little data available concerning the effect of protective surface layers on the mechanical behavior of current high strength columbium alloys. This restricts the design engineer to utilizing possibly erroneous estimates of composite mechanical properties based on data obtained in inert environments with uncoated columbium alloy materials. A recently completed program sponsored by the Bureau of Naval Weapons(1) to investigate the design properties of coated Fansteel 85 alloy evidenced a particularly detrimental influence of protective coatings on the tensile ductility of FS-85 alloy in the temperature range 1200-1600°F. A study is currently being conducted at Solar Aircraft(2) to determine the influence of several protective coating systems on the mechanical behavior of refractory metal foil. This work has revealed widely varying contributions of protective surface layers to the design properties of columbium alloy foil, particularly in altering the substrate tensile properties and in producing a brittle composite fracture behavior.

Elucidating the effect of protective surface layers on the mechanical properties of columbium base materials is imperative if useful coating-base metal systems are to be developed. Guidance is needed in future alloy and coating development efforts to produce coated columbium alloys with useful mechanical properties. The recently concluded Design Data Study on Pfaudler and TRW coated Fansteel 85 alloy has been continued under Bureau of Weapons Contract NOw 63-0471c. The purpose of this program is to investigate the design properties of Pfaudler and TRW coated B-66 and X-110 (D-43) columbium alloys. This program and the testing procedures will be conducted in a manner similar to that of the previous Design Data Study, endeavoring to generate comparative design property data for the six coating-columbium alloy systems. In addition to establishing design criteria for these specific coated refractory metal systems, the development of testing equipment and procedures for the determination of coated refractory metal properties is necessary and is an invaluable contribution to the advancement of refractory metal technology.

2. DESIGN DATA STUDY OF COATED FS-85 ALLOY

The design data study of coated Fansteel 85 alloy provided a comparative evaluation of the mechanical properties of two coating-base metal systems. The following design criteria were investigated for the Pfaudler Cr-Mo modified silicide and the TRW Cr-Ti-Si coatings applied to 30 mil FS-85 alloy sheet:

1. Cyclic oxidation properties in air at 1600 to 2600°F.

2. Thermal shock-erosion-oxidation properties involving thermal shock cycles from 2600° to 250°F followed by post oxidation at 1600°F.

3. Tensile properties of uncoated (vacuum) and coated (air) sheet from room temperature to 2600°F.

4. Tensile prestrain tolerance of the two coatings based on retention of 2 hour oxidation protection at 1600 and 2600°F after tensile prestrain at temperatures from room temperature to 2600°F.

5. Stress rupture properties of the coating-base metal composites in air at 1600 to 2600°F.

The results of these evaluation tests were presented in a final summary report to the Bureau of Weapons(1) at the conclusion of the program.

A very limited follow-up on the design properties of these two coating-base metal systems was conducted in the initial portion of this program. Additional tests were performed in the areas of (1) tensile properties and (2) strain to coating failure to conclude the immediate effort in evaluating the design properties of coated FS-85 alloy.

2.1 Tensile Properties of Coated FS-85 Alloy

The influence of the Pfaudler and TRW coatings on the tensile properties of FS-85 alloy was comparable throughout the temperature range room temperature to 2600°F. From room temperature to 800°F and from approximately 1800 to 2600°F the properties of the uncoated sheet tested in vacuum and the coated sheet tested in air also compared very favorably. However, in the temperature range 800 to 1600°F uncoated FS-85 alloy sheet exhibited a marked susceptibility to strain aging, resulting in a very pronounced peak in the ultimate tensile strength and a corresponding minimum in tensile elongation. This behavior was shown graphically in the final summary report[1] on the previous contract. The application of the protective coatings to FS-85 alloy virtually eliminated the effect of this intermediate temperature strengthening mechanism along with reducing the composite tensile elongation to only 3-5% at 1200-1600°F.

Strain aging is an interstitially dependent strengthening phenomenon characteristic of body centered cubic metals. It is attributed to the obstruction of lattice dislocation motion during deformation by the migration of interstitial atoms to the stress fields of mobile dislocations. Ingram has interpreted the available data on the strain aging behavior of refractory metals in terms of dislocation models proposed by Cottrell[3]. According to his findings oxygen and hydrogen atoms are the diffusing species which are responsible for strain aging in columbium. Apparently the strain aging behavior of the FS-85 alloy rendered the alloy notch sensitive in the 800-1600°F range. Cracks initiated in the brittle coating after a small amount of tensile strain propagated with ease through the coated substrate, resulting in a very low capacity for composite plastic deformation.

The heat of FS-85 alloy sheet (85D-633) used in the previous Design Data Study contained approximately 250 ppm carbon, well in excess of the product specification (100 ppm)[4]. This high level of carbon was not discovered until after the evaluation specimens had been prepared for the mechanical property study, and the schedule of the program would not permit the delay of processing a new heat of material. It was felt, however, that the overall high interstitial level of this heat of material may have been a critical factor in the brittle behavior of coated FS-85 sheet in the 800-1600°F temperature range. An additional quantity of 30 mil FS-85 alloy sheet was, therefore, acquired from the Fansteel Metallurgical Corporation for further tensile property evaluation in this critical temperature range. Table 1 lists the chemical compositions of these two heats of material.

Tensile specimens were prepared from this second heat of material for a determination of both smooth and notched tensile properties at room temperature and 1200°F. Three sheet conditions were evaluated:

1. Uncoated - recrystallized as received.

2. Uncoated - simulated Cr-Ti-Si coating heat treatment.

3. TRW Cr-Ti-Si coated - 8 hours at 2300°F and 4 hours at 2000°F.

TABLE 1

Chemical Analyses of 30 Mil
Fansteel 85 Alloy Sheet

Element	Heat No. 85D-633(1)	Heat No. 85D-691(2)
O	150 ppm	60 ppm
H	-	3 ppm
N	20 ppm	10 ppm
C	250 ppm	40 ppm
Ta	27.92%	27.8 %
W	10.0 %	10.3 %
Zr	0.98%	0.96%
Fe	<0.005%	0.007%
Ni	<0.005%	-
Si	<0.005%	0.01 %
Ti	<0.005%	-
Cb	Balance	Balance

(1) Evaluated in previous contract - NOw 62-0098c

(2) Evaluated in current contract - NOw 63-0471c

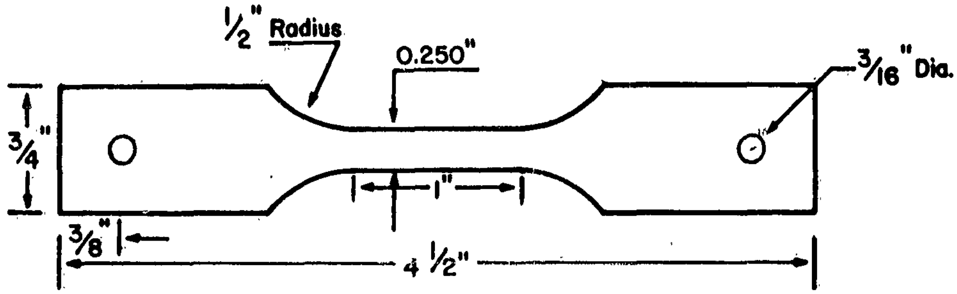
The Pfaudler and TRW coatings displayed equivalent influences on the mechanical behavior of FS-85 alloy in the previous work, therefore, only the TRW coating was used in this limited investigation. Drawings of the smooth and notched specimens are shown as types A and D in Figures 1 and 2. The strain rate used was again 0.020 in/in/min, in order to be consistent with previous testing techniques. Table 2 lists the tensile properties obtained with this second lot of FS-85 sheet. The ultimate strength and elongation values are plotted in Figures 3 and 4 as a comparison with the tensile properties of the higher carbon heat of material. TRW coated specimens remaining from the previous program were also tensile tested as a current comparison of the two material lots.

At room temperature tensile elongation in excess of 20% was obtained with all smooth and coated specimens of both heats. Notch sensitivity of FS-85 alloy was not evident at room temperature, since the notch strength to ultimate strength ratio exceeded one for the two uncoated sheet conditions.

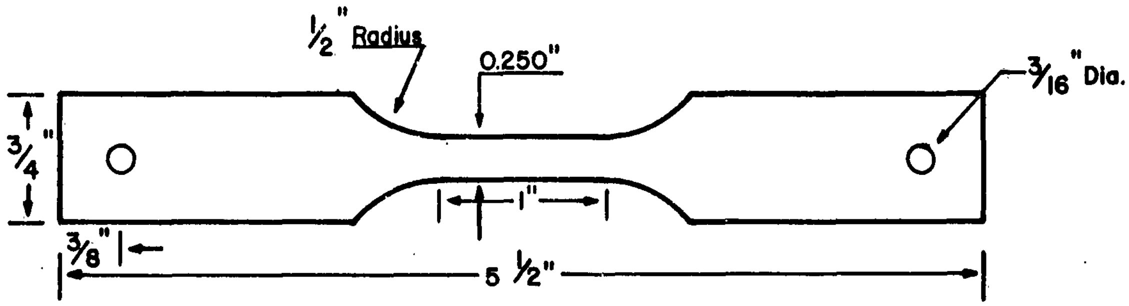
The tensile elongation of uncoated sheet from the second heat was again less at 1200°F than at room temperature, as was the case with the previous material lot. However, as shown in Figure 3, strain age strengthening was considerably less effective with the second lot of FS-85 alloy sheet, as evidenced by the lower ultimate strength at 1200°F. The lower interstitial level of the second heat may account for this reduction in strain aging.

However, the influence of the protective coating on the mechanical properties of the "cleaner" material was again that of eliminating the strain age strengthening and reducing the composite tensile elongation to 4-5% at 1200°F. Even with the much lower oxygen and carbon levels FS-85 alloy was notch sensitive as coated in the intermediate temperature range. The mechanical properties of both heats of coated sheet were comparable at 1200°F.

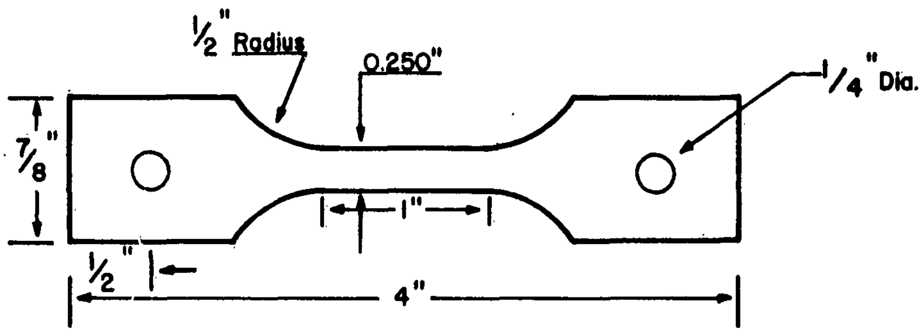
It was found in the previous work with coated FS-85 alloy that removal of the protective coating from the specimen surface completely restored the tensile elongation at 1200°F to a level comparable to that of the uncoated sheet. The built-in notch provided by the coating appeared to be the most critical factor in the composite fracture behavior. However, as shown in Table 2, the notch strength to ultimate strength ratio for the uncoated FS-85 sheet was again greater than one at 1200°F, indicating the substrate was not notch sensitive for this machined notch configuration. The machined notch radius was approximately 0.001 inch. For a shallow elliptical notch in a sheet specimen, the elastic stress



A · TENSILE SPECIMEN - RT to 2300°F

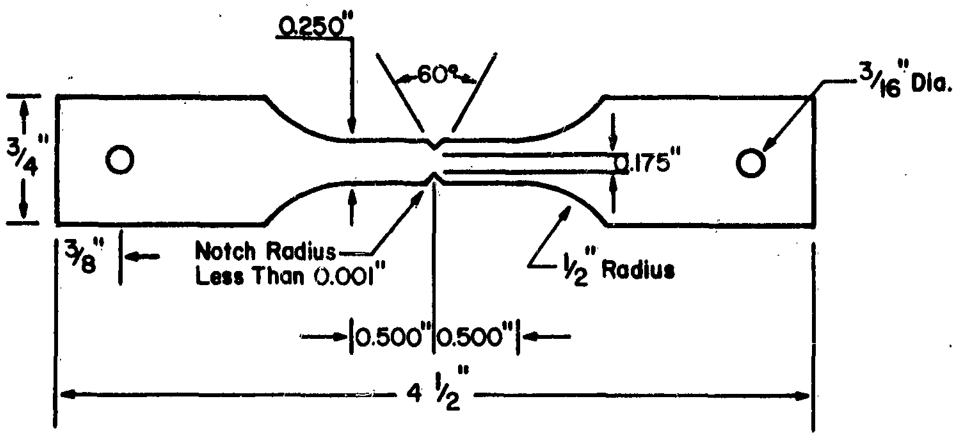


B · TENSILE SPECIMEN - 2500 and 2600°F

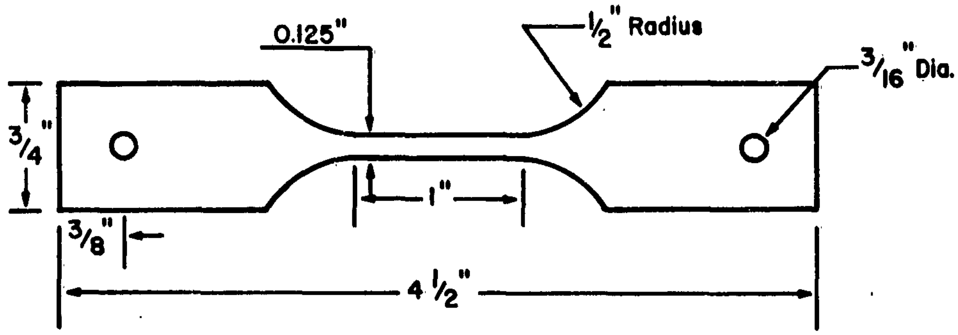


C · CREEP SPECIMEN - 2000 to 2600°F

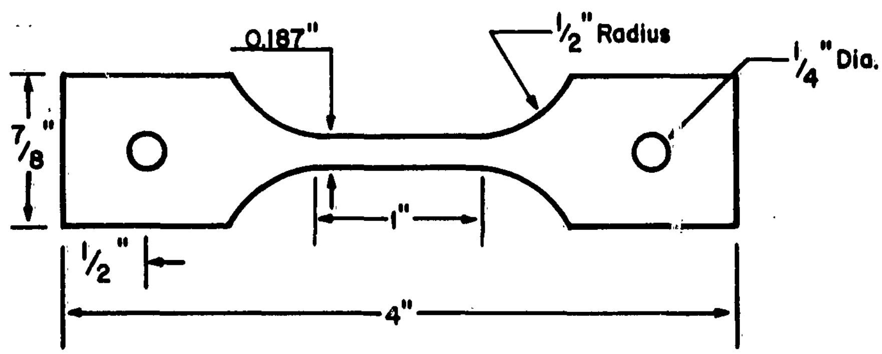
Figure 1 Tensile and Creep Specimen Designs for Mechanical Property Tests



D · NOTCHED TENSILE SPECIMEN



E · PRESTRAIN OXIDATION SPECIMEN



F · CREEP SPECIMEN - 1600°F

Figure 2 Notched Tensile, Prestrain and Creep Specimen Designs for Mechanical Property Tests

TABLE 2

Tensile Properties of Uncoated and Coated 30 Mil FS-85 Alloy Sheet at Room Temperature and 1200°F

Sheet Specimen Condition	Room Temperature Ultimate Strength PSI	Room Temperature 0.2% Offset Yield Strength PSI	Room Temperature Elong. %	Room Temperature Reduction of Area %	1200°F Ultimate Strength PSI	1200°F 0.2% Offset Yield Strength PSI	1200°F Elong. %	1200°F Reduction of Area %
Recrystallized(1)								
Smooth (Average)	86,000	69,500	22.2	52	62,500	42,500	14.2	79
Recrystallized(2)								
Smooth	80,700	58,400	28.0	67.2	48,300	27,600	14.0	35.6
Smooth	80,800	56,300	28.0	64.8				
Notch	85,600	Ns/Us=1.07			50,400	Ns/Us=1.06		
Notch	87,000				52,400			
Cr-Ti-Si Coating Heat Treatment(3)								
Smooth	76,100	56,100	29.0	73.2	48,200	26,400	16.0	53.1
Smooth	76,300	53,600	29.0	75.4	45,900	24,700	17.0	53.7
Notch	84,400	Ns/Us=1.11			49,700	Ns/Us=1.06		
Notch	84,800				49,400			
Preoxidized-Smooth(4)					38,300	24,200	5.0	21.0
Smooth					39,200	25,900	4.5	9.5
As Cr-Ti-Si Coated(5)	73,500	56,500	23.8	73.1	35,000	28,200	4.7	0.0
As Cr-Ti-Si Coated	75,200	56,300	24.5	78.4	34,800	28,500	4.5	0.0
As Cr-Ti-Si Coated(6)	77,600	64,000	26.1	68.6	38,100	31,600	5.3	0.4
As Cr-Ti-Si Coated	81,400	65,800	26.7	64.1	38,700	31,000	3.8	0.0

(1) Heat No. 85D-633 - Recrystallized - 1 hour 2300°F - Vacuum
(2) Heat No. 85D-691 - As received - recrystallized.
(3) Heat No. 85D-691 - Heat treated 8 hours at 2300°F plus 4 hours at 2050°F in vacuum
(4) Heat No. 85D-691 - Preoxidized 1 hour in an argon + air atmosphere
(5) Heat No. 85D-691 - TRW Cr-Ti-Si coated
(6) Heat No. 85D-633 - TRW Cr-Ti-Si coated
(7) Strain rate 0.020 in./in./min. to fracture

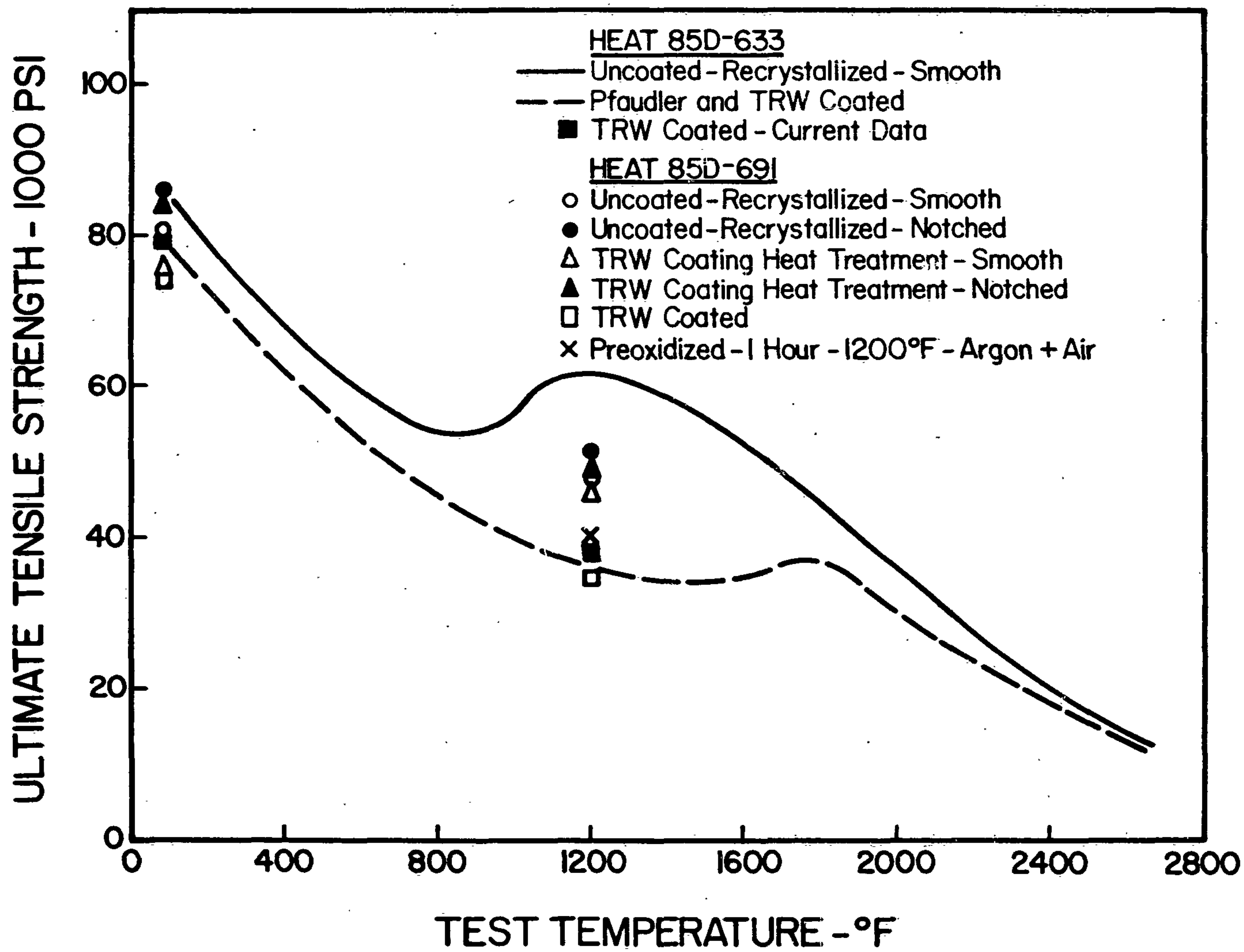


Figure 3 Ultimate Tensile and Notch Strength of Uncoated and Coated FS-85 Alloy Sheet As a Function

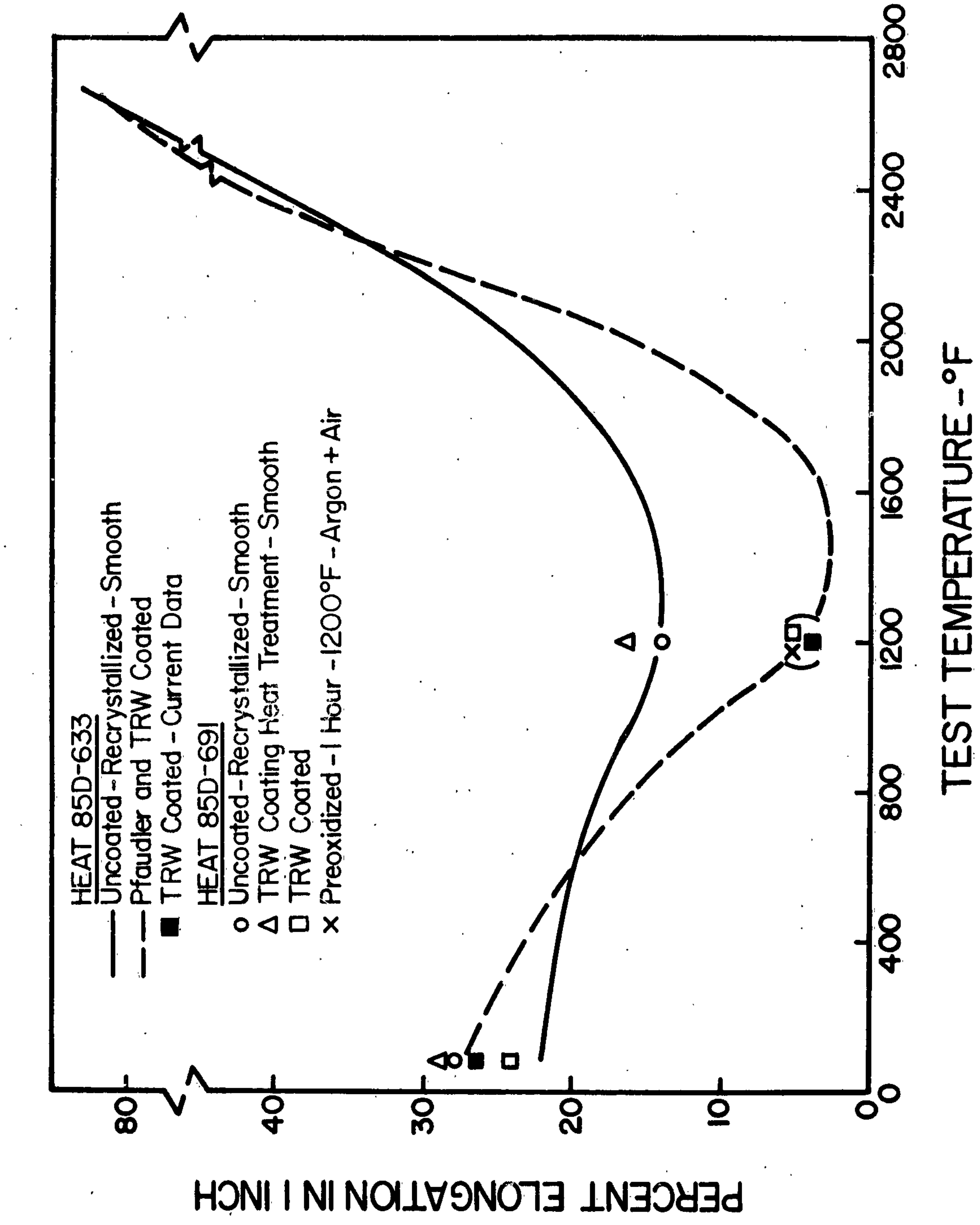


Figure 4 Tensile Elongation of Uncoated and Coated FS-85 Alloy Sheet As a Function of Test Temperature

concentration factor according to Neuber is given by:

$$K_{t,s} = 1+2\sqrt{t/r}$$

where t is the notch depth and r is the notch radius[5]. An approximate stress concentration factor of 13 can be calculated for the notch machined in these sheet specimens. Note that for a sharp crack, where the notch radius approaches zero, the stress concentration factor based on this relationship would approach infinity.

Localized yielding at the base of the machined notch apparently prevented crack initiation in the uncoated FS-85 notched specimens. The built-in notch effect created by cracks in the coatings was therefore not simulated. Other factors obviously play a role in the fracture behavior of coated specimens, such as the compositional gradient across the coating and into the substrate. Also, there is the relative susceptibility of the substrate to the continuation of kinetic crack propagation as opposed to crack initiation solely within the substrate at the base of a notch.

The critical dependence of notch sensitivity on the radius at the notch base was further substantiated by the tensile properties of preoxidized uncoated FS-85 specimens tested at 1200°F. Two uncoated as received smooth tensile specimens were inadvertently oxidized in an argon + air atmosphere for 1 hour at 1200°F prior to testing at this temperature. The properties of these two specimens, as shown in Table 2, were comparable to those of the coated sheet. It was metallographically observed in a cross section of one of these specimens that many sharp cracks were initiated in the brittle subscale on the specimen surface during tensile loading. The nature of these sharp cracks corresponded very closely to the cracks produced in the brittle protective coatings during tensile testing.

It is evident that there is a need for an evaluation of the notch properties of existing and future columbium base alloys in this intermediate temperature strain aging range.

2.2 Strain to Coating Failure

An important design property of a coating-refractory metal system is the tolerance of the coating for deformation without the loss of coating protection. A series of tests were conducted in the previous program to evaluate the prestrain tolerances of Pfaudler and TRW coatings on FS-85 alloy. This involved prestraining coated tensile type specimens at temperatures from room temperature to 2600°F, followed by 2 hours of oxidation at both 1600 and 2600°F. Specimens were deformed to increasing levels of total strain (elastic and plastic) until coating failure was observed within the 2 hour exposure. Two hours was arbitrarily selected as the exposure time to indicate gross cracking as a result of prestrain.

In order to very briefly investigate the effect of prestrain on the ultimate protective lives of the Pfaudler and TRW coatings, cyclic oxidation was continued at 1600 and 2600°F with prestrained specimens on which coating failure had not previously been observed after 2 hours of exposure. This data is presented in Table 3. Cyclic oxidation was terminated after 50 hours at 1600°F and 60 hours at 2600°F because of gross oxidation propagating from coating failures in deformed pinholes in specimen grip sections.

Each of the specimens on which cyclic oxidation was continued was prestrained to a level just below the established prestrain tolerance, but beyond the elastic limit of the coated substrate. It can be observed that for post oxidation at 2600°F the protective lives of the Pfaudler and TRW coatings were comparable in both the prestrained and as coated conditions. However, several prestrained specimens on which coating failure was not observed within 2 hours at 1600°F then failed in 26-44 hours of continued cyclic oxidation at this temperature. At 1600°F, where the selfhealing capabilities of the coatings are ineffective, cracks initiated in the outer coating layers by prestraining apparently continue to propagate during thermal cycling, eventually resulting in coating failure. As the exposure temperature increases both selfhealing and the increased plasticity of the coatings reduce the susceptibility of the coatings to oxidation failure in a region of previously induced micro-cracks. These data indicate that the prestrain tolerance of a coating-base metal system lies within a narrow region of deformation in which cracks are initiated in the coatings and rapidly propagate into the ductile substrate.

3. DESIGN DATA STUDY FOR COATED B-66 AND X-110 ALLOYS

Previous studies have shown that many of the protective coatings applicable to columbium alloys have characteristically different influences on the mechanical behavior of the coating-substrate composites. However, it would obviously not be practical or economically feasible to evaluate the properties of all the prospectively useful protective coating-columbium alloy systems. Prior to the previous Design Data Study a series of comparative evaluation tests were conducted with D-14 and FS-85 alloys to screen the currently promising protective coatings available for columbium alloys.

TABLE 3

Oxidation Protective Lives of Prestrained
Pfaudler and TRW Coated FS-85 Alloy
Tensile Type Specimens

Prestrain Temperature °F	Average Proportional Limit % Strain(1)	Prestrain Level % Total Strain(2)	Cyclic Post Oxidation Protective Life-Hour			
			TRW Coating		Pfaudler Coating	
			1600°F	2600°F	1600°F	260
RT	2.2	2.5	-	-	50*(3)	-
		2.8	-	-	-	3F(
		4.1	-	60*	-	-
		6.3	50*	-	-	-
800	1.6	0.9	-	-	-	7F
		2.3	-	-	38F	-
		3.9	-	60*	-	-
		7.4	26F	-	-	-
1200	1.4	1.5	-	-	50*	-
		2.0	-	-	-	3F
		4.1	44F	60*	-	-
1600	1.1	1.3	-	-	-	3F
		1.5	-	-	38F	-
		2.0	50*	60*	-	-
		No Prestrain (average)	>150	>50	>150	5

(1) Proportional limit - obtained from load extension curve plotted by Instron machine
(2) Total strain - elastic + plastic
(3) * Denotes no evidence of coating failure in prestrained gage section - tests discontinued
(4) F denotes failure in prestrained gage section

The Pfaudler Cr-Mo modified silicide and the TRW Cr-Ti-Si diffusion alloy coatings were selected from this analysis and subsequently employed in the determination of the design properties of coated FS-85 alloy. Since the completion of these screening tests there have been no notable improvements reported for any of these protective coating systems; therefore, additional screening studies for the present program were not considered necessary.

Selection of the sheet alloys for the second Design Data Study involved the consideration of three columbium base sheet materials recently recommended for further development by the Refractory Metal Sheet Rolling Panel of the Materials Advisory Board: X-110 (Cb-10W-1Zr), B-66 (Cb-5Mo-5V-1Zr) and Cb-752 (Cb-10W-2.5Zr). Fansteel 85 is the fourth columbium alloy in this category. The Westinghouse B-66 alloy and the Du Pont X-110 alloy were selected from this group as the two sheet materials for the mechanical property study. Due to the similar nominal composition of X-110 and Cb-752 alloys, comparable behavior of the two alloys in the coated condition would be expected. Orders have been placed for 2100 square inches of 30 mil sheet of each alloy and delivery is anticipated late in May.

Figure 5 is a flow chart showing the proposed course of the program. Each of the outlined tasks is then briefly discussed in the subsequent sections.

3.1 Proposed Preliminary Evaluation Tests

A brief series of preliminary evaluation tests are planned to investigate both the possibility of anomalous protective behavior of the Pfaudler and TRW coatings on B-66 and X-110 alloys, and also the tensile ductility of the two coated alloys in the 1200°F temperature range where coated FS-85 alloy exhibited a sharp minimum in tensile elongation. Very briefly, these tests will involve evaluation of the following:

(1) Bend transition temperature of recrystallized uncoated sheet.

(2) Room temperature bend ductility of as coated sheet.

(3) Room temperature bend ductility of coated sheet after post oxidation for 100 hours at 2000°F and 20 hours at 2600°F.

(4) Cyclic oxidation of coated coupons at 1600, 2000, 2300 and 2600°F.

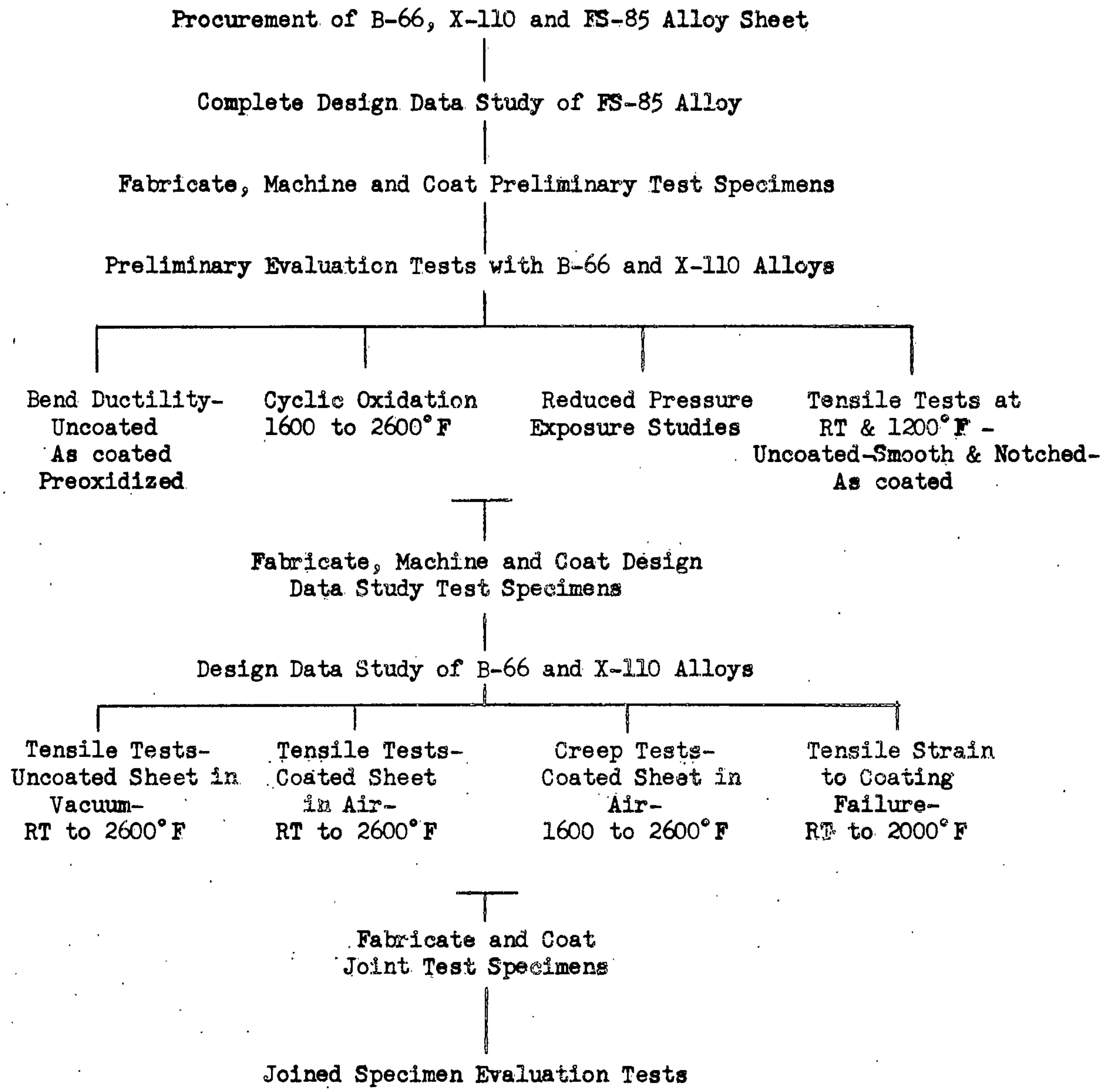


Figure 5 Flow Chart for Design Data Study Program

(5) The influence of low pressure - high temperature exposure on the subsequent one atmosphere protective life of the two coatings.

(6) Smooth and notch tensile properties of uncoated and coated sheet at room temperature and 1200°F.

These tests will be conducted during preparation of the larger number of test specimens for the major portion of the program.

3.1.1 Bend Ductility Tests

The brittle-to-ductile bend transition temperature of both B-66 and X-110 alloy sheet will be determined for sheet in the recrystallized condition. All bend testing will be conducted according to specifications recommended by the Refractory Metal Sheet Rolling Panel of the Materials Advisory Board(6).

Pfaudler and TRW coated sheet will be room temperature bend tested to assess the effect of the coatings and coating processing treatments on the bend ductility of the coated substrate at the normal handling temperature.

The effectiveness of the two coatings in deterring the penetration of embrittling interstitials into the columbium substrate will be evaluated in a third series of bend tests. Coated specimens will be exposed in air under cyclic conditions for 100 hours at 2000°F and 20 hours at 2600°F and then room temperature bend tested. Retention of substrate bend ductility at the normal handling temperature will be the criterion for acceptable protection.

3.1.2 Cyclic Oxidation

Cyclic oxidation tests will be conducted on 1/2" x 1/2" coated coupons of both alloys in air at one atmosphere to ascertain the level of protection which might be anticipated for the two coatings during the subsequent mechanical property tests. Previous studies have shown that the protective life of these coatings is greater in static exposure, even under stress, than when exposed under thermal cycling conditions. Specimens representing all four coating-base metal systems will be exposed simultaneously at 1600, 2000, 2300 and 2600°F using globar heated furnaces. Cyclic oxidation will consist of air cooling the specimens to room temperature once each hour for eight cycles, followed by approximately 16 hours of non-cyclic exposure in each 24 hour period. This procedure will be continued until there is visible evidence of substrate attack.

3.1.3 Low Pressure Exposure

Almost all evaluation studies of protective coatings for columbium base alloys rely on the behavior of the coatings in air at one atmosphere as the measure of protective capability. However, utilization of protective coated columbium alloy materials in aerospace re-entry type vehicles, in applications such as heat shields or leading edge components, will involve high temperature exposure in very low pressure oxidizing environments. It is of utmost importance that a correlation between the gas-coating reaction kinetics and the stabilities of these coatings at one atmosphere and at low pressures be established. Attention is being given to this problem in other programs.[7, 8] Only a very limited effort is planned for this program, merely to demonstrate by a few selected tests, whether serious degradation is suffered by the Pfaudler and TRW coatings on B-66 and X-110 alloys as a result of elevated temperature-low pressure exposure. Coated specimens will be exposed in air at 2500°F, employing two reduced pressures and two periods of exposure time. The selection of the specific test conditions will be based on prior studies of the TRW Cr-Ti-Si coating tested in the same low pressure facility. Cyclic oxidation of the pre-exposed specimens at one atmosphere at both 2000 and 2600°F will then be employed to evaluate the effect of the reduced pressure exposure on the protective life of the two coatings.

Bend tests will be conducted with similar coated specimens which have also been exposed under both the low pressure and subsequent one atmosphere oxidation conditions. Although it is unlikely that substrate embrittlement would occur in the static low pressure environment, chemical changes will undoubtedly occur in the coatings during this reduced pressure exposure. Enhanced oxygen or nitrogen diffusion through the coatings as a result of compositional alterations could lead to substrate embrittlement during subsequent one atmosphere oxidation.

3.1.4 Tensile Properties

In view of the detrimental effect of the Pfaudler and TRW coatings on the tensile ductility of FS-85 alloy in the temperature range 1200-1600°F, tensile tests will be conducted at room temperature and 1200°F with both the B-66 and X-110 alloys to investigate the possibility of a similar behavior. As discussed in Section 2, the notch sensitivity of coated FS-85 alloy was enhanced in the 1200°F temperature range by a strain aging phenomenon, and crack propagation from tensile cracks in the brittle coatings produced brittle fracture of the coating-base metal composites. By removing the protective coatings from the specimen surface ductility was restored to FS-85 alloy, comparable to that obtained with recrystallized uncoated sheet. In order to study the susceptibility of B-66 and X-110 alloy sheet to this type of embrittlement, both alloys will be tensile tested at room temperature and 1200°F in three basic conditions:

1. Uncoated recrystallized sheet - smooth and notched specimens.

2. Uncoated sheet subjected to simulated coating heat treatments - smooth and notched specimens.

3. Pfaudler and TRW coated sheet - smooth specimens.

Specimen types A and D shown in Figures 3 and 4 are the sheet specimen designs which will be used in these tests. If brittle behavior does exist, these test conditions may permit an evaluation of whether crack propagation from the built-in notch provided by the coatings is intensified by the diffusion of coating elements into the columbium substrate, the coating process heat treatment, the strain aging mechanism or interactions between these factors. In the event the machined notch is again not sharp enough to simulate the coating cracks, a fatigue or preoxidation technique will be utilized with a limited number of specimens to obtain a notch with a very sharp radius.

3.2 Proposed Design Data Study

As pointed out in the flow chart in Figure 5, four principal areas will be investigated in the Design Data Study with Pfaudler and TRW coated B-66 and X-110 alloys:

1. Tensile properties of uncoated sheet (vacuum) and coated sheet (air) from room temperature to 2600°F.

2. Tensile prestrain tolerance of the protective coatings after elastic and plastic composite deformation, based on retention of 2 hour post oxidation protection in air at 1600 and 2600°F.

3. Creep deformation properties of the protective coating-base metal composites in air at 1600, 2000, 2300 and 2600°F.

4. Fabrication, coating and evaluation of simple joint test specimens and small joined assemblies.

The alterations which were necessary in the test equipment and evaluation procedures utilized for these tests in the previous Design Data Study are discussed in the following sections. The test specifications recommended by the Refractory Metal Sheet Rolling Panel of the Materials Advisory Board(6) will be employed in these tests where applicable.

3.2.1 Tensile Tests

The tensile properties of uncoated B-66 and X-110 alloys will be determined in vacuum at temperatures from room temperature to 2600°F. Only recrystallized sheet will be tested in this program since it will generally be necessary to design on the basis of recrystallized sheet properties. Most coating processing treatments and the majority of the proposed application environments involve exposures above the recrystallization temperatures of these columbium base materials.

Tensile test data are available from the producers of B-66 and X-110 alloys, and are plotted along with the properties of FS-85 alloy as a function of the test temperature in Figures 6 through 10. Based on these data for uncoated recrystallized sheet, the Westinghouse B-66 alloy is the strongest of the three columbium base materials both in terms of actual tensile and yield strengths and on a strength to weight basis. The Du Pont X-110 alloy shows nearly comparable strength above 2000°F, however, insufficient data was reported to graphically characterize the alloy properties in the intermediate temperature range.

A very pronounced strain aging peak is exhibited by both the FS-85 and B-66 alloys in the 800-1600°F temperature range, corresponding to an increase in the ultimate tensile and yield strengths and a minimum in the alloy ductility. Based on the similarities in mechanical behavior of these three columbium alloys, notch sensitivity and susceptibility to brittle fracture in this temperature range are anticipated for both the coated B-66 and X-110 alloys.

The coated tensile specimens will again be tested in air utilizing the infrared radiant heating facility constructed for the previous program. The specimen designs which will be used in these tensile tests and in all of the design property evaluations are shown in Figure 2.

3.2.2 Prestrain Tolerance

From a design standpoint, one of the most important properties of a coating-base metal system is the tolerance of the protective

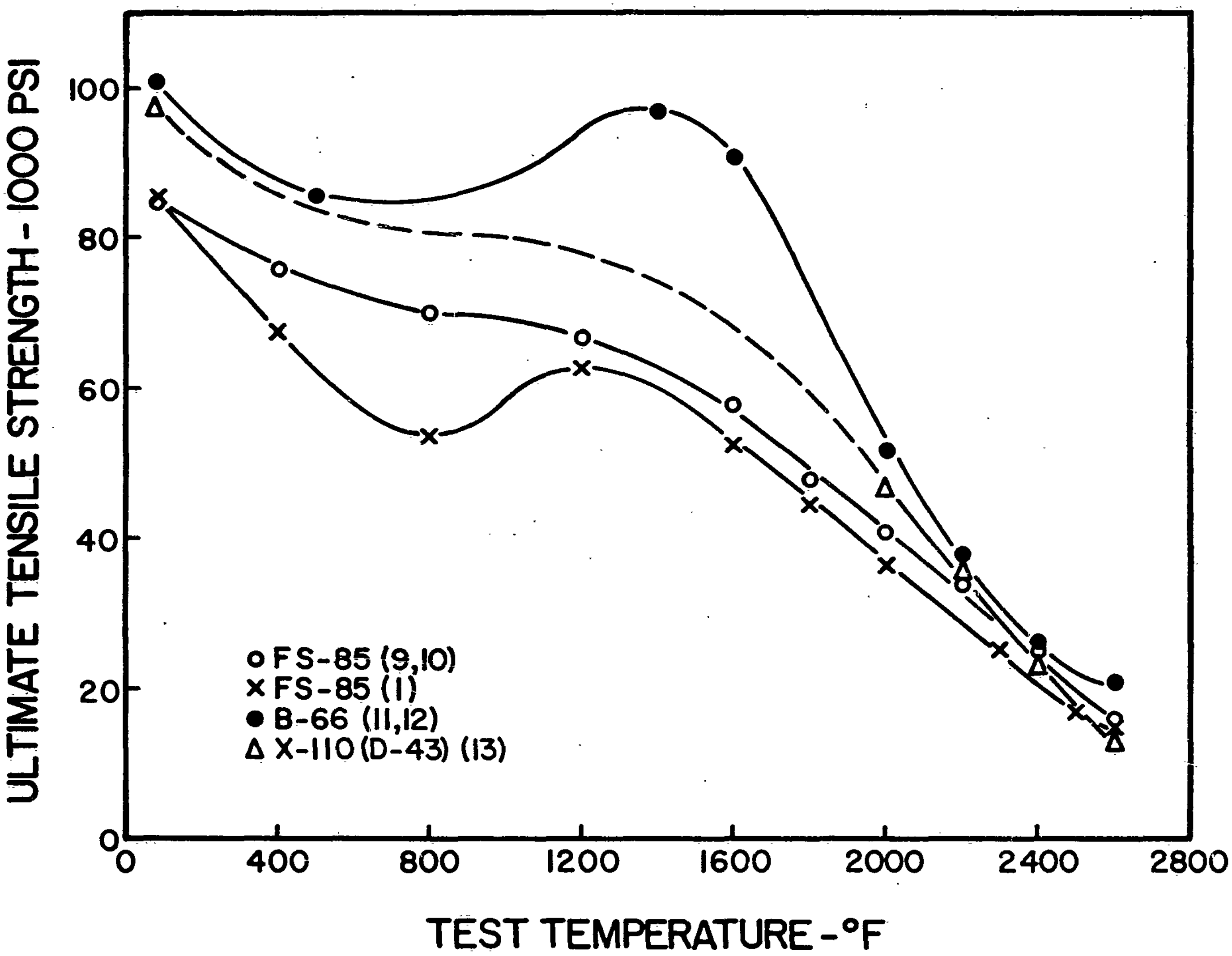


Figure 6 Ultimate Tensile Strength As a Function of Test Temperature for Uncoated Recrystallized FS-85, B-66 and X-110 Columbium All

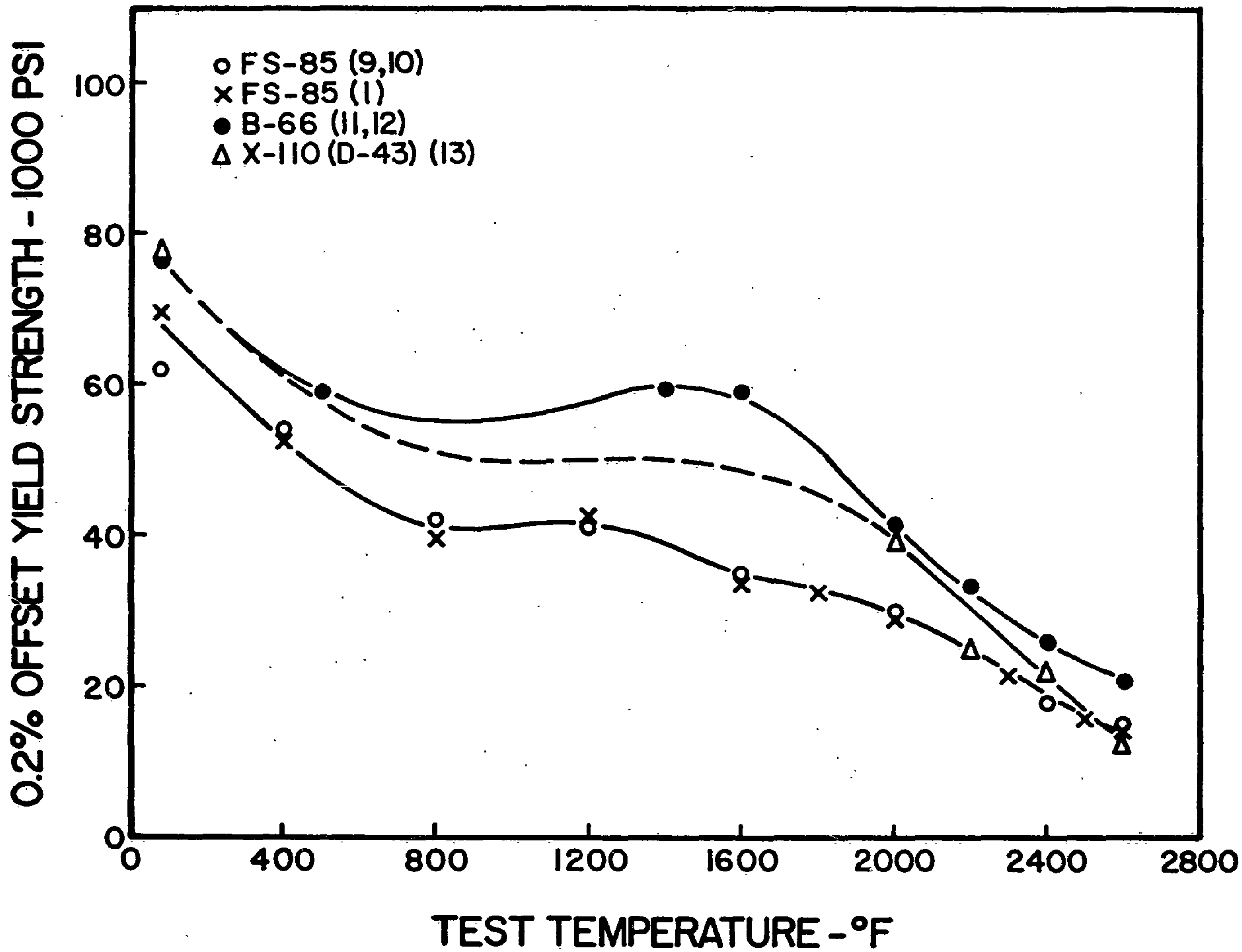


Figure 7 Yield Strength As a Function of Test Temperature for Uncoated Recrystallized FS-85, B-66 and X-110 Columbium Alloys

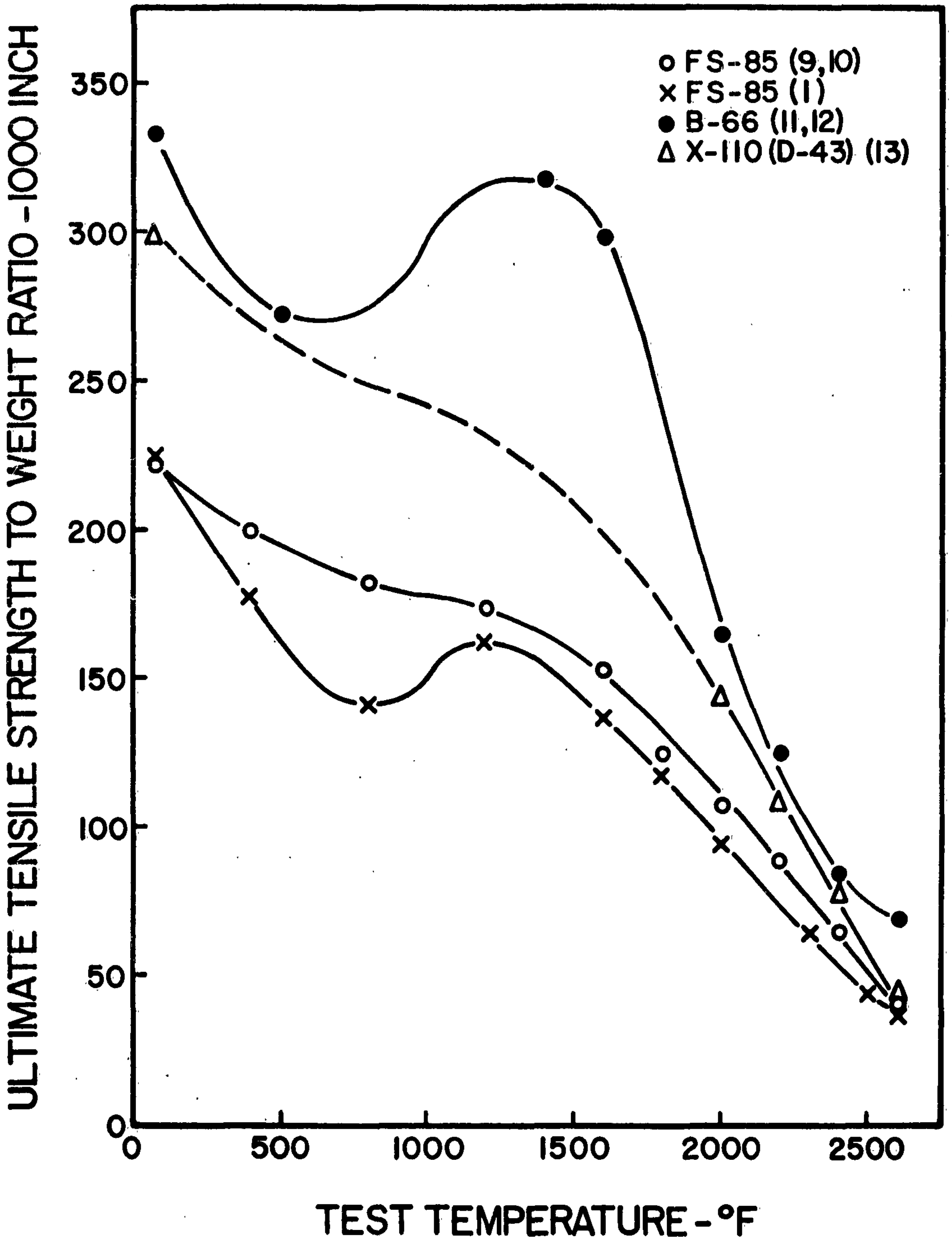


Figure 8 Ultimate Tensile Strength to Weight Ratio As a Function of Test Temperature for Uncoated Recrystallized FS-85, B-66 and X-110 Columbium Alloys

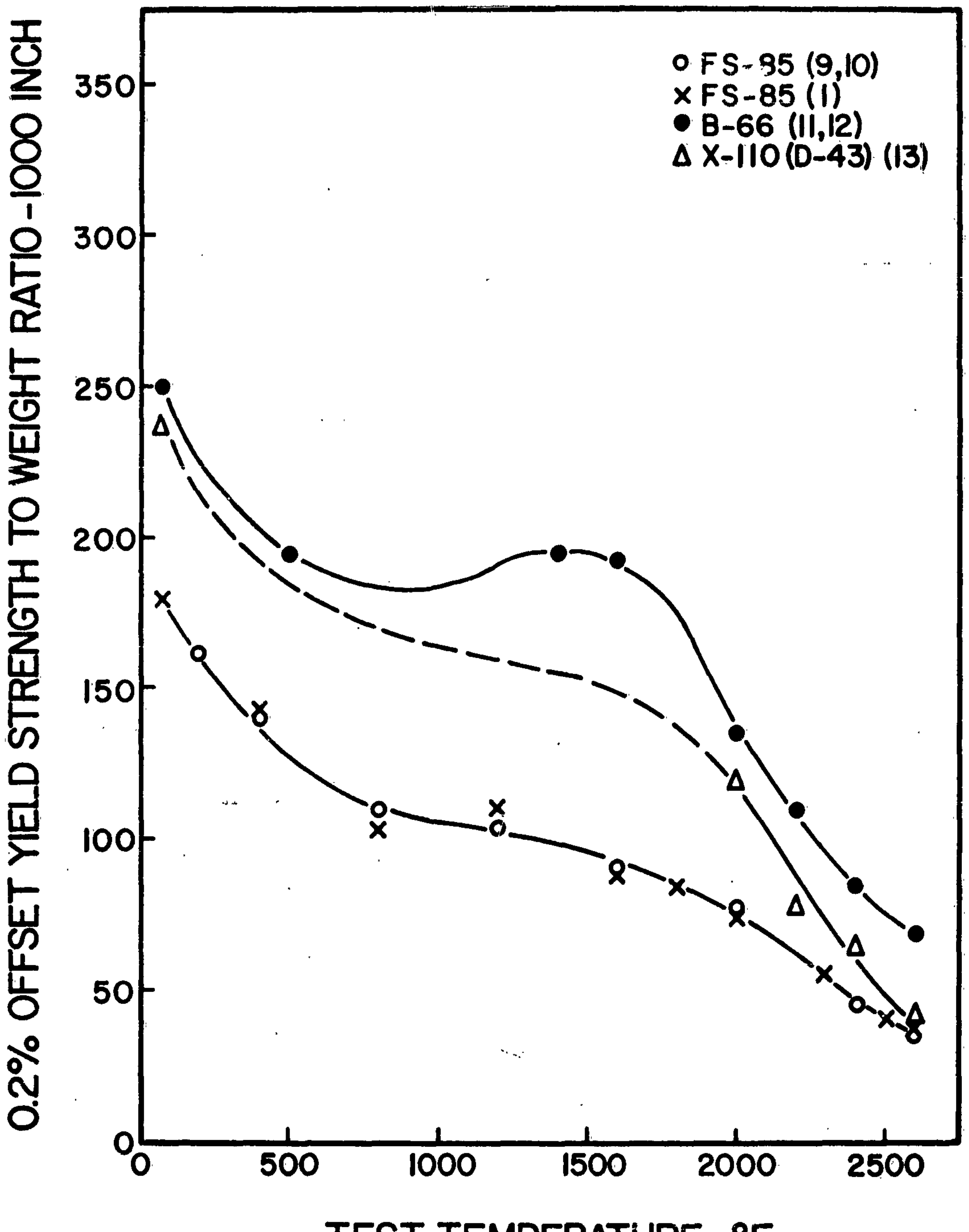


Figure 9 Yield Strength to Weight Ratio As a Function of Test Temperature for Uncoated Recrystallized FS-85, B-66 and X-110 Columbium Alloys

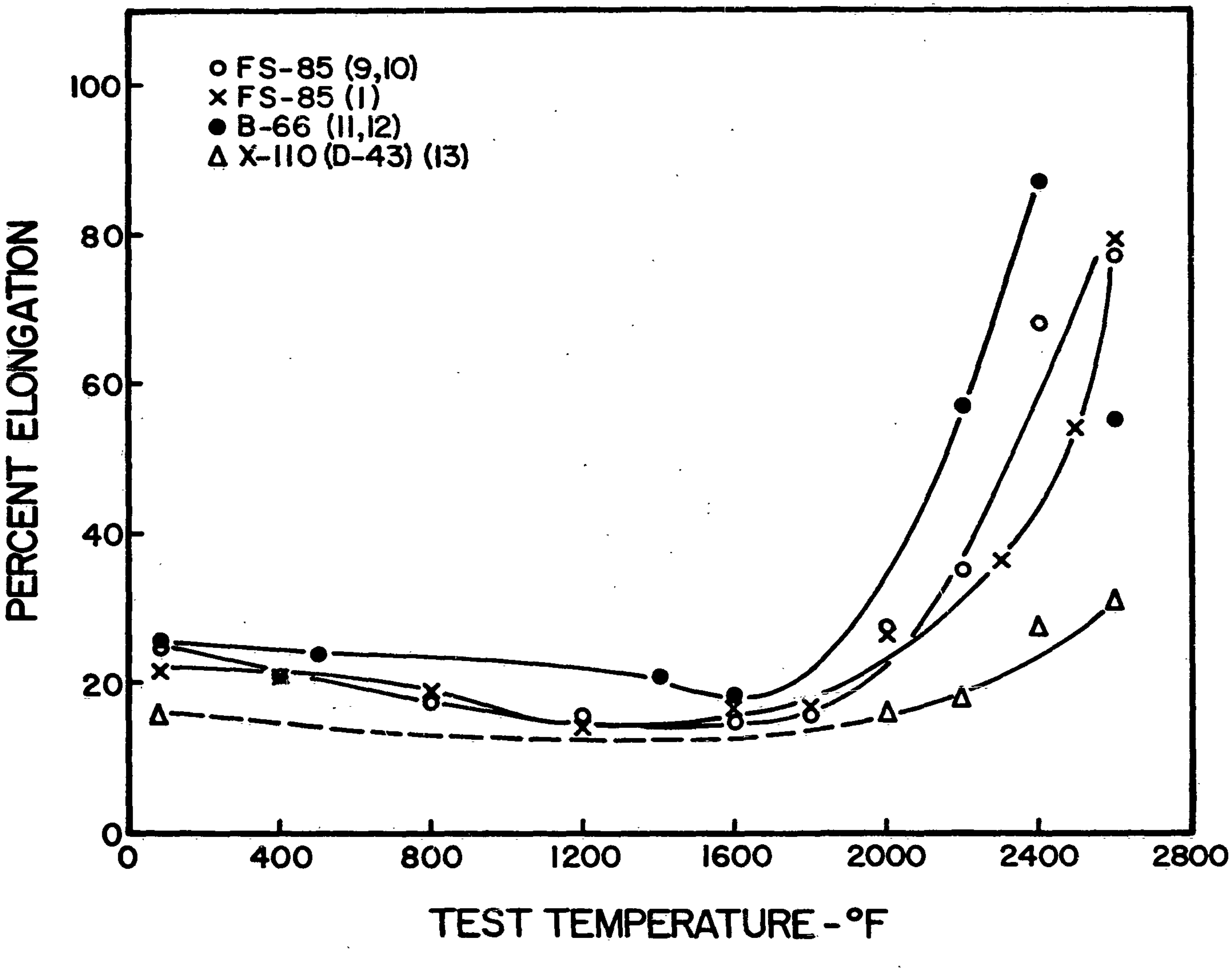


Figure 10 Tensile Elongation As a Function of Test Temperature for Uncoated Recrystallized FS-85, B-66 and X-110 Columbium Alloys

coating for composite deformation without the loss of coating protective capability. In the series of tests designed to study this property coated specimens will be prestrained in tension to increasing levels of total strain (elastic + plastic) and subsequently oxidation tested for 2 hours in air at 1600 and 2600°F. The absence of substrate attack after the 2 hour post oxidation treatment will constitute prestrain within the deformation tolerance. Two hour protection was arbitrarily selected as a convenient measure of the absence of severe crack propagation through the coatings as a result of the applied stress. The 1600°F post oxidation treatment represents exposure at a temperature below which the selfhealing capabilities of these coatings are effective, while 2600°F is within the range where selfhealing is expected.

An attempt will be made in this series of tests to analyze the phenomenon of crack initiation and propagation in these coating-base metal systems. The influence of strain rate on the deformation tolerance will be investigated for prestraining at room temperature. Low and intermediate strain rates will be obtained using Baldwin and Instron tensile machines and a very high strain rate will be achieved by employing a ballistic impact device.

The specimen design which was utilized in the previous program to tensile prestrain coated FS-85 alloy was Type A shown in Figure 1. An insufficient differential in load bearing area existed between the pinhole and gage cross section in this specimen design. This resulted in inconsistencies in the degree of prestrain deformation occurring in these two locations on each individual specimen. The total specimen strain was continuously plotted by the Instron tensile machine in terms of crosshead extension. Poor correlation between the crosshead extension and strain in the specimen gage section, because of pinhole deformation, rendered termination of prestrain at a predetermined level virtually impossible. The actual prestrain level was determined by measuring the plastic strain in the specimen gage section using a comparator; and then adding this value to the elastic strain as indicated by the load-extension plot. Reduction of the specimen gage width, as shown in Type E of Figure 2, should provide an adequate area differential to assure that deformation occurs primarily in the specimen gage section.

3.2.3 Creep Properties

As discussed previously[1] the 30-50% creep exhibited by all Pfaudler and TRW coated FS-85 alloy specimens prior to rupture at 2000°F and above virtually eliminates the utility of stress rupture data as design criteria for these coating-base metal systems. From a design standpoint, creep behavior within design limits is a far more useful property of a prospective structural material. Creep studies will therefore be conducted with Pfaudler and TRW coated B-66 and X-110 alloys in air at 1600, 2000, 2300 and 2600°F, within the limits of 5% total creep and 150 hours of stress oxidation exposure. Various levels of stress and creep time will be investigated at the four temperatures such that sufficient data is generated to graphically delineate the creep characteristics of the four coating-base metal systems. Correlations can then be made between the relative creep rates of the four systems and the tolerances of the coatings for creep deformation without loss of oxidation protection.

As pointed out previously(1), specimen pinhole failures frequently occurred in the stress rupture tests of coated FS-85 alloy at 1600 and 2600°F. At 1600°F pinhole fracture was attributed to creep deformation in this area of stress concentration, ultimately exceeding the deformation tolerance of the coatings and resulting in gross oxidation of the columbium substrate. Since modification of the specimen grip section poses many difficult coating protection problems, the alternative of reduction in the specimen gage width will be employed to reduce the necessary applied load and correspondingly the stress level in the grip section. This specimen is shown as Type F in Figure 2.

At 2600°F failure in the specimen grip section was of a catastrophic nature, resulting from failure of the protective coatings and melting of the substrate oxidation products. The melting reaction was attributed to the proximity of the specimen grip sections to the furnace heating elements, forcing the grip sections to operate at temperatures considerably in excess of the 2600°F test temperature. Modifications were made in rebuilding this furnace involving enlargement of the furnace hearth and relocation of the heating elements such that all points on the test specimens will be equidistant from the element surfaces. Five independent creep tests may be conducted simultaneously in this facility.

3.2.4 Evaluation of Coated Joints

The successful coating and protection of joint configurations will necessarily play a vital role in furthering the advancement of coated refractory metals as high temperature materials of construction. Numerous problems confront the coater of refractory metal joint assemblies, in addition to those inherent to the coating process. In the case of a riveted joint design, consideration must be given to the order of operations such as fabrication, joint assembly, upsetting of the rivets and application of the protective coating. The throwing power of the coating process and degradation of the joint mechanical properties must be considered with all joint designs. Compatibility of the protective coatings with braze materials is still another area to be investigated.

This final portion of the Design Data Study will involve the fabrication, coating and evaluation of several simple joint test specimens. Design of the test configurations and development of evaluation techniques are not yet complete, however, the study will be limited to riveted, spot welded and fusion welded joints with Pfaudler and TRW coated B-66 and X-110 alloys.

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Prepared by: J. D. Gadd
J. D. Gadd, Research Metallurgist
Materials Processing

Prepared by: D. B. Warmuth
D. B. Warmuth, Metallurgist
Materials Processing

Approved by: R. A. Jefferys
R. A. Jefferys, Research Supervisor
Materials Processing

Approved by: A. S. Nemy
A. S. Nemy, Manager
Materials Processing

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